

(21) Application No. 55675/73 (22) Filed 30 Nov. 1973 (19)

(23) Complete Specification filed 29 Nov. 1974

(44) Complete Specification published 17 Aug. 1977

(51) INT. CL.² C01B 31/26 17/14

(52) Index at acceptance

CIA E10A E2K2 K9B3B K9B6A2 K9B6AY K9B6X
K9B6Y K9BY

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(54) IMPROVEMENTS IN OR RELATING TO A PROCESS FOR THE
MANUFACTURE OF CARBON DISULPHIDE

(71) We, COURTAULDS LIMITED, a British Company, of 18, Hanover Square, London, W1A 2BB, England, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The invention is concerned with a process for the manufacture of carbon disulphide.

Carbon disulphide can be made by the reaction of a hydrocarbon, usually methane, with sulphur. The reaction is carried out continuously, generally in a reactor tube at a temperature of 450 to 700°C. Sulphur is preferably used in higher than stoichiometric amounts, so that the reaction product comprises carbon disulphide and hydrogen sulphide together with unreacted sulphur, typically about 10 per cent by weight of the sulphur used. The carbon disulphide and hydrogen sulphide are separated from the sulphur by cooling and condensing the sulphur. The sulphur is recovered and recycled to the reactor feed. The sulphur is preferably kept liquid between recovery and recycling to allow easy transport.

The above recycling procedure has the disadvantage that the sulphur contains solid impurities such as metal sulphides and carbon compounds, typically about 0.2 per cent by weight of the sulphur. These impurities are carried back into the reactor system and some are deposited in the reactor tube, thus building up a deposit which necessitates a shutdown of the reactor for cleaning.

We have found that filtration of the untreated recovered sulphur is not practicable because of entrained combustible gases in the sulphur. These gases consist mainly of carbon disulphide and hydrogen sulphide with a small amount of the hydrocarbon, e.g. methane.

Accordingly the invention provides a continuous process for the manufacture of carbon disulphide by the reaction of a hydrocarbon and sulphur in which unreacted sul-

phur is recovered from the reaction product and recycled, wherein the recovered sulphur is treated in the liquid phase to remove entrained combustible gases by passing an inert gas through it and the treated sulphur is filtered to remove solid impurities before being recycled.

By an inert gas we mean a gas which is inert to liquid sulphur and to carbon disulphide and hydrogen sulphide, as well as to the hydrocarbon, e.g. methane, used in the reaction. The preferred inert gas is steam. Nitrogen is an alternative.

The invention will now be described, by way of example, with reference to the accompanying drawing, in which:

Figure 1 is a flow sheet of one embodiment of the invention, and

Figure 2 is a diagrammatic section of one example of an apparatus suitable for carrying out the degassing step of the invention.

Referring to Figure 1 of the accompanying drawing, sulphur vapour and methane are fed to a reactor 1. This reactor consists of a reactor tube kept at a temperature of from 450 to 700°C in a furnace. The reaction product is cooled and passes to a sulphur condenser 2, where water is used as a cooling medium to condense the sulphur, and thence to a separation system 3, where the sulphur is removed from the gaseous product. The sulphur is recovered in liquid form, and passes to a degassing apparatus 4 and filter press 5. It is then recycled to the sulphur feed to the reactor at a point before the sulphur is vaporised.

The degassing apparatus 4 is shown in Figure 2. A container 10 is divided into three compartments 11, 12, 13 interconnected by passages 14, 15. In each compartment are steam sparging pipes 16 having apertures such as 17 along their length. Each compartment is also provided with unapertured heating coils such as 18 fed with superheated steam to ensure that the sulphur is kept liquid. The apparatus is provided with an inlet 19 through which the impure

20 sulphur, containing carbon disulphide, hydrogen sulphide, methane, and solid impurities, enters. An exit 20 is provided for the sulphur which has been stripped of its
5 entrained gases by the steam sparging. A vent pipe 21 is provided for the steam to-
gether with the carbon disulphide, hydrogen sulphide and methane stripped from the sul-
phur. The gases can be recovered from the
10 steam and treated to recover carbon disulphide and hydrogen sulphide (for conversion of sulphur) in conventional manner.

We have found that recovered sulphur containing 0.2 *per cent* solid impurities
15 which has been stripped of gases in this manner can be satisfactorily filtered, and a reduction of the solid impurities level to 20 parts per million has been achieved using two filtering steps.

20 Although the invention has been exemplified by reference to one specific degassing apparatus, other conventional degassing apparatus, such as a column stripper, can alternatively be used.

25 WHAT WE CLAIM IS:—

1. A continuous process for the manu-
30 facture of carbon disulphide by the reaction of a hydrocarbon and sulphur in which unreacted sulphur is recovered from the re-

action product and recycle, wherein the recovered sulphur is treated in the liquid phase to remove entrained combustible gases by passing an inert gas through it and the
35 treated sulphur is filtered to remove solid impurities before being recycled.

2. A process according to claim 1 wherein the inert gas is steam.

3. A process according to claim 1 or 2
40 wherein the removal of entrained combustible gases is carried out in apparatus substantially as described with reference to Figure 2 of the accompanying drawings.

4. A process according to any of claims
45 1 to 3 wherein the hydrocarbon reacted with sulphur is methane.

5. A continuous process for the manu-
50 facture of carbon disulphide by the reaction of a hydrocarbon and sulphur substantially as herein described with reference to Figure 1 of the accompanying drawings.

6. Carbon disulphide when manufac-
55 tured by a process according to any of claims 1 to 5.

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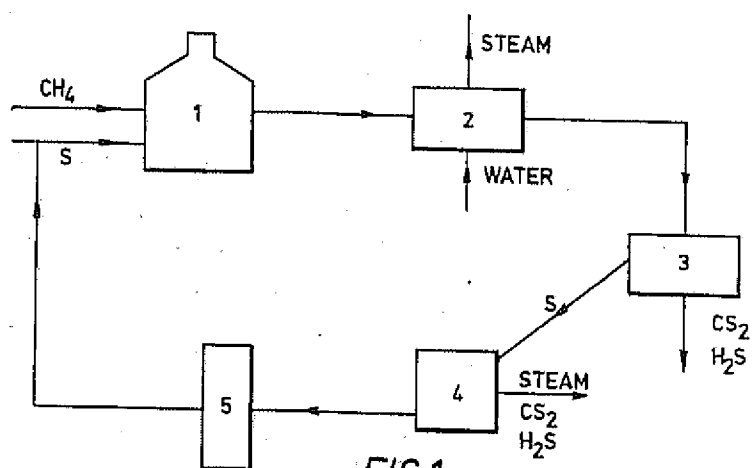


FIG. 1.

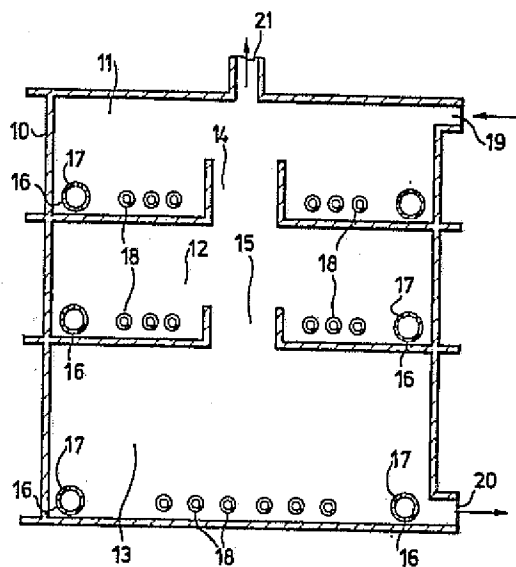


FIG. 2.

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PROCESS FOR THE MANUFACTURE OF CARBON DISULPHIDE

Publication number: GB1483193

Publication date: 1977-08-17

Inventor:

Applicant: COURTAULDS LTD

Classification:

- international: **C01B31/26; C01B31/00**; (IPC1-7): C01B31/26; C01B17/14

- European:

Application number: GB19730055675 19741129

Priority number(s): GB19730055675 19741129

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Abstract of **GB1483193**

1483193 Carbon disulphide; purifying sulphur COURTAULDS Ltd 29 Nov 1974 [30 May 1973] 55675/73 Heading C1A In a continuous process for the manufacture of carbon disulphide by the reaction of a hydrocarbon, e.g. methane and sulphur in which unreacted sulphur is recovered from the reaction product and recycled, the recovered sulphur is treated in the liquid phase to remove entrained combustible gases by passing an inert gas (e.g. steam or nitrogen) through it and the treated sulphur is filtered to remove solid impurities before being recycled.

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Description of **GB1483193**

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British Company, of 18, Hanover Square,

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the sulphur used. The carbon disulphide and hydrogen sulphide are separated from the sulphur by cooling and condensing the sulphur. The sulphur is recovered and recycled to the reactor feed. The sulphur is preferably kept liquid between recovery and recycling to allow easy transport.

The above recycling procedure has the disadvantage that the sulphur contains solid impurities such as metal sulphides and carbon compounds, typically about 0.2 per cent by weight of the sulphur. These impurities are carried back into the reactor system and some are deposited in the reactor tube, thus building up a deposit which necessitates a shutdown of the reactor for cleaning.

We have found that filtration of the untreated recovered sulphur is not practicable because of entrained combustible gases in the sulphur. These gases consist mainly of carbon disulphide and hydrogen sulphide with a small amount of the hydrocarbon, e.g.

methane.

Accordingly the invention provides a continuous process for the manufacture of carbon disulphide by the reaction of a hydrocarbon and sulphur in which unreacted sulphur is recovered from the reaction product and recycled, wherein the recovered sulphur is treated in the liquid phase to remove entrained combustible gases by passing an inert gas through it and the treated sulphur is filtered to remove solid impurities before being recycled.

By an inert gas we mean a gas which is inert to liquid sulphur and to carbon disulphide and hydrogen sulphide, as well as to the hydrocarbon, e.g. methane, used in the reaction. The preferred inert gas is steam.

Nitrogen is an alternative.

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The degassing apparatus 4 is shown in Figure 2. A container 10 is divided into three compartments 11, 12, 13 interconnected by passages 14, 15. In each compartment are steam sparging pipes 16 having apertures such as 17 along their length. Each compartment is also provided with unapertured heating coils such as 18 fed with superheated steam to ensure that the sulphur is kept liquid. The apparatus is provided with an inlet 19 through which the impure sulphur, containing carbon disulphide, hydrogen sulphide, methane, and solid impurities, enters. An exit 20 is provided for the sulphur which has been stripped of its entrained gases by the steam sparging. A vent pipe 21 is provided for the steam together with the carbon disulphide, hydrogen sulphide and methane stripped from the sulphur. The gases can be recovered from the steam and treated to recover carbon disulphide and hydrogen sulphide (for conversion of sulphur) in conventional manner.

We have found that recovered sulphur containing 0.2 per cent solid impurities which has been stripped of gases in this manner can be satisfactorily filtered, and a reduction of the solid impurities level to 20 parts per million has been achieved using two filtering steps.

Although the invention has been exemplified by reference to one specific degassing apparatus, other conventional degassing apparatus, such as a column stripper, can alternatively be used.

WHAT WE CLAIM IS:

1. A continuous process for the manufacture of carbon sulphide by the reaction of a hydrocarbon and sulphur in which unreacted sulphur is recovered from the reaction product and recycle, wherein the recovered sulphur is treated in the liquid phase to remove entrained combustible gases by passing an inert gas through it and the treated sulphur is filtered to remove solid impurities before being recycled.
2. A process according to claim 1 wherein the inert gas is steam.
3. A process according to claim 1 or 2 wherein the removal of entrained combustible gases is carried out in apparatus substantially as described with reference to Figure 2 of the accompanying drawings.
4. A process according to any of claims 1 to 3 wherein the hydrocarbon reacted with sulphur is methane.
5. A continuous process for the manufacture of carbon sulphide by the reaction of a hydrocarbon and sulphur substantially as herein described with reference to Figure 1 of the accompanying drawings.
6. Carbon disulphide when manufactured by a process according to any of

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Claims of **GB1483193**

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claims 1 to 5.

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